

REMARKS

The Office action rejects claims 11 – 27, citing 35 U.S.C §103(a); US 4,344,846 to Klein (hereinafter, “**Klein**”); US 3,958,023 to Butterworth (hereinafter, “**Butterworth**”); and BASF’s 60th Anniversary of Povidone (hereinafter, “**Anniversary**”).

The claims of the present application employ (or are directed to) polymer powders comprising (a) at least one thermoplastic from the group consisting of polyolefins and polyamides, and (b) at least one further substance selected from the group consisting of silicates, carbonates, oxides, silica gel, kieselguhr, diatomaceous earth and crosslinked polyvinyl lactams, and mixtures thereof. In response to a restriction requirement, applicants have elected as (a), polyolefins, and as (b), crosslinked polyvinyl lactams.

The Office action asserts **Klein** discloses the use of polystyrene as a filtration aid. This assertion is not relevant. The enclosed pages from Hawley’s Condensed Chemical Dictionary, Eleventh Edition should make clear, as expressed in reply to the previous Office action, polystyrene is not a polyolefin. Polystyrene is not a polyamide either, but, as noted above, the claims have been restricted to polyolefins as component (a). Applicants respectfully note, however, column 1, lines 12 – 16 of **Klein** states, “[t]he invention is that of (a) a filtration method using as a filter aid micro-bits of an expanded (and non-brittle in its expanded form) thermoplastic polymer selected from a styrene-polymer and a polyolefin from polyethylene to polymethylpentene, and (b) filter aid mixtures of these micro-bits and any of the inorganic filter aids (e.g. diatomaceous earth, perlite or silica).”

Furthermore, the Office action asserts **Butterworth** discloses the use of PVPP admixed with conventional filter aids to treat liquids. The claims,

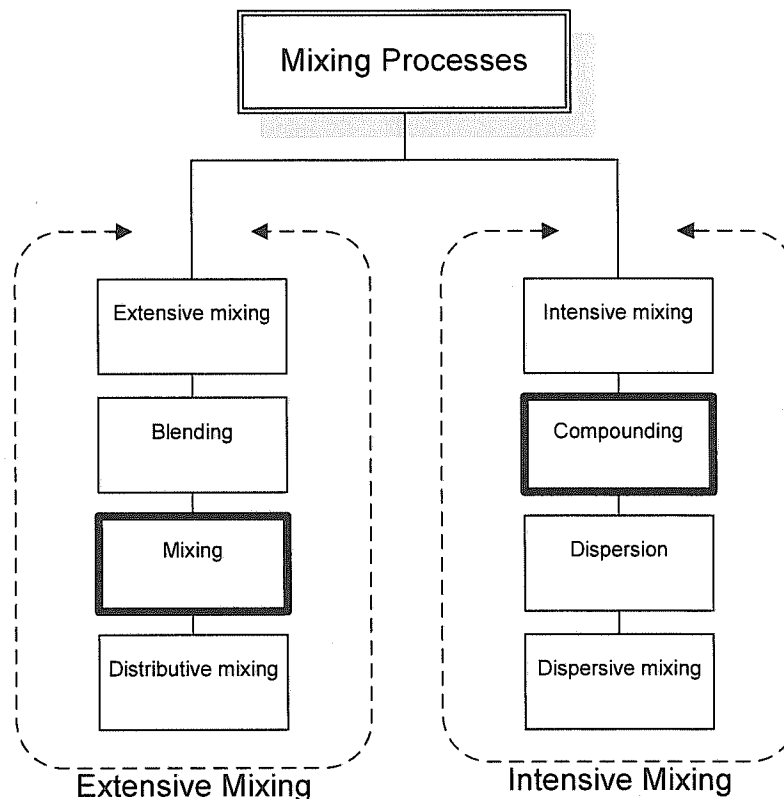
however, employ crosslinked polyvinylactams. **Butterworth** does not teach the use of crosslinked PVPP. This claim feature seems to have been overlooked.

Finally, with respect to claim 11 (and the claims that depend from claim 11), the Office action fails to address the requirement that a suspension must be passed through a porous filter medium at a constant flow rate.

For at least these reasons, a *prima facie* case of obviousness has not been established.

The Office action alleges the specification equates the terms “mixing” and “compounding.” Applicants respectfully submit this allegation is in error. The portions of the specification cited on pages 5 and 7 of the Office action do not equate the terms. The specification uses the terms in harmony with the substantive evidence of record, which indicates compounding is divergent and fundamentally different, even on the most basic level, from admixing.

Chapman and Hall, Morton-Jones, David H., *Polymer Processing*, chapter 3.3, pages 59 – 60, 1989, New York explains “two basic mixing functions” can be identified in the polymer mixing art. The two basic mixing functions are identified as “extensive mixing” and “intensive mixing.” As illustrated in the diagram below, “[e]ach of the two functions, rather confusingly, carries a number of names.” Extensive mixing is also referred to as blending, mixing and distributive mixing. Intensive mixing is also referred to as compounding, dispersion, and dispersive mixing. The following diagram illustrates this nomenclature.



The term, “compounding,” refers to intensive mixing. Sections 3.3.1 and 3.3.2 of the Morton-Jones publication explain the characteristics of extensive mixing and intensive mixing, respectively. Notably, extensive mixing “consists essentially of stirring together the ingredients” and “[t]he result is a mixture of powders; the individual powders remain and can in principle, be separated....” On the other hand, intensive mixing “involves the more intimate dispersion of the additives into the matrix of the polymer.” Intensive mixing “usually requires a physical change in the components [and] ... the polymer to be in the molten or rubbery state during mixing.” On page 60 of the Morton-Jones publication, the resulting granules are characterized as having the same disperse composition after compounding.

Chapman and Hall, Berins, Michael L. (editor), *Plastics Engineering*

Handbook of the Society of the Plastics Industry, chapter 22, page 635, 1991, New York, also explains “[t]he process by which ingredients are intimately melt-mixed together into as nearly a homogeneous mass as is possible is known as compounding.” On pages 645 – 646, the reference, further explains,

- “[s]trictly speaking, compounding involves the fusion of different materials into a homogeneous mass, uniform in composition and structure;”
- in compounding, “the more mobile materials are locked up inside a tough, viscous, doughy envelope;” and
- “[t]he use of heat, either internally generated by shear or externally applied is needed to obtain compound uniformity.”

Still further, the Declaration under 37 C.F.R. §1.132, executed July 12, 2006, evidences that compounding is fundamentally different from admixing. In the declaration,

The sedimentation behavior of the following polymer powders in water was compared:

- A) polystyrene (standard polystyrene PS 158K ground to a mean particle size of $D(v, 0.5) = 196 \mu\text{m}$)
- B) polyvinylpolypyrrolidone (Divergan[®] F, BASF Aktiengesellschaft, mean particle size of $D(v, 0.5) = 27 \mu\text{m}$)
- C) A mixture of polystyrene A and polyvinylpolypyrrolidone B in a ca. 2:1 weight ratio
- D) A compound (mean particle size of $D(v, 0.5) = 45 \mu\text{m}$) of polystyrene and polyvinylpolypyrrolidone in a ca. 2:1 weight ratio

The mean particle size of $D(v, 0.5)$ presents the mean diameter of the smallest 50 % (by volume) fraction of particles which are dispersed in water. The declaration explains that only compound D provides suitable properties.

The declaration makes clear that a physical mixture comprising polystyrene and PVPP is divergent and fundamentally different from a compounded polymer composition comprising polystyrene and PVPP.

The substantive evidence of record indicating that compounding is divergent and fundamentally different, even on the most basic level, from admixing is entirely uncontroverted.

As expressed in the Office action, **Butterworth** discloses the use of PVPP admixed with conventional filter aids to treat liquids

Butterworth also mentions “blending” at column 3, line 42, which, as evidenced above, is another type of extensive mixing. **Butterworth** does not teach any type of intensive mixing. Instead, **Butterworth** teaches a type of mixing that is divergent and fundamentally different from compounding.

“A reference may be said to teach away when a person of ordinary skill, upon reading the reference ... would be led in a direction divergent from the path that was taken by the applicant.” *In re Icon Health and Fitness, Inc.*, 496 F.3d 1374, 1381 (Fed.Cir.2007), citing *In re Gurley*, 27 F.3d 551, 553, 31 USPQ2d 1130, 1131 (Fed.Cir.1994). The U.S. Supreme Court approvingly cited *United States v. Adams*, 383 U.S. 39, 51-52, 86 S.Ct. 708, 15 L.Ed.2d 572 (1966), for the “principle that when the prior art teaches away from combining certain known elements, discovery of a successful means of combining them is more likely to be nonobvious.” *KSR International Co. v. Teleflex, Inc.*, 550 U.S. 398, 416, 127 S.Ct. 1727, 1739-40 (2007).

In light of the uncontroverted evidence of record, **Butterworth** teaches a type of mixing that is so divergent and fundamentally different from compounding that a person having ordinary skill in the art would be led in a direction divergent from the path that was taken by applicants. Such a

teaching is a teaching away, which is important evidence of nonobviousness. Applicants respectfully submit that in the context of **Butterworth**, i.e., chill haze stabilization of aqueous liquids derived from fruits and vegetables, the above-mentioned teaching away cannot be over emphasized.

At column 1, lines 13 – 16, **Butterworth** explains, “[v]arious liquids derived from vegetables and fruits contain various phenolic substances, such as tannins and anthocyanogens, proteins, and compounds or complexes of two or more of these substances.” At column 1, lines 25 – 30, **Butterworth** goes on to explain “relatively high molecular weight compounds which escape filtration can combine after bottling ... to form complex molecules that cause a haze to form in the beverage, particularly when the beverage is chilled. This haze is called chill haze and is an undesirable characteristic in any beverage.” At column 1, lines 30 – 36, **Butterworth** explains, “[i]t is well known in the art to treat beverages derived from vegetables or fruits with conventional absorbents [*sic*] or coagulants to adsorb, or coagulate and precipitate, the undesirable tannins, proteins, or combinations thereof from the beverage before the beverage is bottled.”

McGraw-Hill, Concise Encyclopedia of Science and Technology, Third Edition, page 27 (Sybil P. Parker, ed., McGraw-Hill 1994), describes “adsorption,” indicating interfacial contact between two immiscible phases is needed. *Ullmann’s Encyclopedia of Industrial Chemistry*, Fifth Edition, Volume A28, pages 56 – 59 (Barbara Elvers, & Stephen Hawkins, eds. VCH Publishers, New York 1996), describes “coagulation,” indicating, especially at section 7.4 on page 58, particle collisions are needed.

According to **Butterworth** chill haze agents adsorb or coagulate and precipitate undesirable compounds. Either mechanism requires direct contact between the chill haze agent and the undesirable compound.

Therefore, a person having ordinary skill in the art would not have diverged from **Butterworth**'s teaching to admix or blend a chill haze agent and a filter aid so as to employ a fundamentally different type of mixing, such as compounding. Again, Chapman and Hall, Berins, Michael L. (editor), *Plastics Engineering Handbook of the Society of the Plastics Industry*, chapter 22, pages 645 – 646, explains, “compounding involves the fusion of different materials into a homogeneous mass, uniform in composition and structure;” and in compounding, “the more mobile materials are locked up inside a tough, viscous, doughy envelope.”

Therefore, at the time the present invention was made, a person having ordinary skill in the art would not have had a reasonable expectation of successfully practicing **Butterworth**'s invention after diverging from **Butterworth**'s teaching to admix so as to employ a fundamentally different type of mixing, such as compounding. The evidence of record does not establish that a person having ordinary skill in the art could have reasonably predicted whether sufficient direct contact between a chill haze agent and undesirable compounds would occur to facilitate adsorption or coagulation and precipitation of the undesirable compounds after the chill haze agent was compounded with a filter aid.

Furthermore, MPEP §2143.01 explains, “[t]he mere fact that references can be combined or modified does not render the resultant combination obvious, unless the results would have been predictable to one of ordinary skill in the art. Of course, “if a technique has been used to improve one device, and a person of ordinary skill in the art would recognize that it would improve similar devices in the same way, using the technique is obvious unless its actual application is beyond his or her skill.” *KSR v. Teleflex*, 550 U.S. at 401.

Anniversary mentions a technique, which seems to have been used to improve delivery of certain drugs. Specifically, **Anniversary** mentions, “a patent-protected novel pharmaceutical manufacturing technology: drug is incorporated by melt extrusion in a matrix consisting of a pharmaceutical polymer.” **Anniversary** focuses on a particular pharmaceutical polymer, indicating, “[d]ue to its thermoplasticity and balanced aqueous solubility properties, Kollidon grades have been found to provide a comprehensive and universal base for various types of drugs.” **Anniversary** explains, “[a]fter melt extrusion, the active drug can be present in the extrudate in one or two forms: as a crystal suspended in the hardened Kollidon matrix, or as a molecule dissolved in the polymer during the melting phase and remaining dissolved in the finished product – a ‘solid solution.’”

No evidence of record establishes that the **Butterworth** invention is similar, in any way, to the pharmaceutical manufacturing technology described in **Anniversary**. No evidence of record establishes that a person of ordinary skill in the art would recognize that the **Anniversary** technique would improve the **Butterworth** invention in the same way. In fact, as discussed above, the uncontroverted evidence of record establishes that **Butterworth** taught away from the **Anniversary** technique and that a person having ordinary skill in the art had no reasonable way to predict whether sufficient direct contact between a chill haze agent and undesirable compounds would occur to facilitate adsorption or coagulation and precipitation of the undesirable compounds if the chill haze agent was compounded with a filter aid.

Furthermore, applicants respectfully submit column 1, lines 12 – 18, **Klein** describes,

(a) a filtration method using as a filter aid micro-

bits of an expanded (and non-brittle in its expanded form) thermoplastic polymer selected from a styrene-polymer and a polyolefin from polyethylene to poly-methylpenten, and (b) filter aid mixtures of these micro-bits and any of the inorganic filter aids (e.g. diatomaceous earth, perlite or silica).

At column 1, lines 53 – 60, **Klein** describes these **expanded** micro-bits by stating, “[t]he nature and characteristics of the vinyl-polymer micro-bits used in this filtration method are described in [U.S. Pat. No. 4,207,378]....”

At column 1, lines 17 – 30, the ‘378 patent explains, the

expanded styrene-polymer or expanded polyolefin microbits ... (i) range in size from about 325 micros in length and also from possibly about the same in width down to about 40 microns or less in length and to about 20 microns or less in width, (ii) have a specific gravity of from about 85 percent, to about the same as that, of the starting polymer which was expanded and then disintegrated into the micro-bits, (iii) are from substantially completely to entirely completely free of intact cells of the expanded polymer from which they were produced and (iv) their particles lack uniformity in outline.

A person having ordinary skill in the art would not expect **Klein’s** expanded micro-bits to retain any of these characteristics upon being compounded with crosslinked PVPP. After all, the uncontroverted evidence of record establishes, at least: (1) compounding involves the fusion of different materials into a homogeneous mass, uniform in composition and structure (2) in compounding, the more mobile materials are locked up inside a tough, viscous, doughy envelope, and (3) the use of heat, either internally generated by shear or externally applied is needed to obtain compound uniformity.

Since a person having ordinary skill in the art would not expect **Klein's** expanded micro-bits to retain their characteristics upon being compounded with crosslinked PVPP, the person having ordinary skill in the art could not have reasonably predicted whether the compounded polymer composition would be useful as a filter aid.

For at least these reasons, applicants respectfully request reconsid

Regarding Official Notice

Applicants respectfully reassert the traversal of the Official Notice statements made in reply to the previous Office action. The Official Notice statements should be withdrawn.

Petition for Extension of Time

Applicants respectfully request that a two-month extension of time be granted in this case. The respective fee is paid by credit card.

Fee Authorization

Please charge any shortage in fees due in connection with the filing of this paper, including any shortage in Extension of Time fees, to Deposit Account 14.1437. Please credit any excess fees to such account.

Conclusion

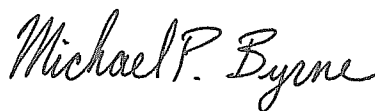
The present application is in condition for allowance, and applicants respectfully request favorable action. In order to facilitate the resolution of

any questions, the Examiner is welcome to contact the undersigned by phone.

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Respectfully submitted,
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Enclosure (3):

- Sax, N. Irving, ed., *Hawley's Condensed Chemical Dictionary*, Van Nostrand Reinhold, New York (1987), pages 940 and 943.
- *McGraw-Hill, Concise Encyclopedia of Science and Technology*, Third Edition, page 27 (Sybil P. Parker, ed., McGraw-Hill 1994); and
- *Ullmann's Encyclopedia of Industrial Chemistry*, Fifth Edition, Volume A28, pages 56 – 59 (Barbara Elvers, & Stephen Hawkins, eds. VCH Publishers, New York 1996).

Hawley's Condensed Chemical Dictionary

ELEVENTH EDITION

Revised by

N. Irving Sax

and

Richard J. Lewis, Sr.



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polymer, stereospecific. (stereoregular).

A polymer whose molecular structure has a definite spatial arrangement, i.e., a fixed position in geometrical space for the constituent atoms and atomic groups comprising the molecular chain, rather than the random and varying arrangement that characterizes an amorphous polymer. Achievement of this specific steric (three-dimensional) structure (also called tacticity) requires use of special catalysts such as those developed by Ziegler and Natta about 1950. Such polymers are wholly or partially crystalline. Synthetic natural rubber, cis-polyisoprene, is an example of a stereospecific polymer made possible by this means. There are five types of stereospecific (or stereoregular) structures: cis, trans, isotactic, syndiotactic, and tritactic.

See also catalyst, stereospecific.

polymer, syndiotactic. See syndiotactic polymer.**polymer, synthetic.** See polymer.

polymer, water-soluble. Any substance of high molecular weight that swells or dissolves in water at normal temperature. These fall into several groups, including natural, semisynthetic, and synthetic products. Their common property of water solubility makes them valuable for a wide variety of applications as thickeners, adhesives, coatings, food additives, textile sizing, etc.

See specific entries.

(1) *Natural*. This type is principally comprised of gums, which are complex carbohydrates of the sugar group. They occur as exudations of hardened sap on the bark of various tropical species of trees. All are strongly hydrophilic. Examples are arabic, tragacanth, karaya.

(2) *Semisynthetic*. This group (sometimes called water-soluble resins) includes such chemically treated natural polymers as carboxymethylcellulose, methylcellulose, and other cellulose ethers, as well as various kinds of modified starches (ethers and acetates).

(3) *Synthetic*. The principal members of this class are polyvinyl alcohol, ethylene oxide polymers, polyvinyl pyrrolidone, polyethyleneimine.

polymethacrylate resin. See acrylic resin, methyl methacrylate.**polymethylbenzene.** See durene and pseudocumene, the two members of this group with some commercial production and use.**polymethylene polyphenylisocyanate.**

A polymer of diphenylmethane-4,4' diisocyanate.

polymethylene wax. See wax, polymethylene.**poly-4-methylpentene-1.**

Properties: High resistance to all chemicals except carbon tetrachloride and cyclohexane, excellent heat resistance, high clarity and light transmittance. Temperature limit 170°C, d 0.83.

Use: Laboratory ware (beakers, graduates, etc.), electronic and hospital equipment; food packaging, especially types subject to high temperature such as trays for TV dinners, etc.; light reflectors.

poly(methyl vinyl ether). See polyvinyl methyl ether.**polymorphism.** See allotropy.

polymyxin. CAS: 1406-11-7. Generic term for a series of antibiotic substances produced by strains of *Bacillus polymyxa*. Various polymyxins are differentiated by the letters A, B, C, D, and E. All are active against certain gram-negative bacteria. Polymyxin B is most used.

Properties: All are basic polypeptides, soluble in water; the hydrochlorides are soluble in water and methanol, insoluble in ether, acetone, chlorinated solvents, and hydrocarbons. Permissible food additives.

Use: Medicine (antibiotic), beer production.

polynuclear. Descriptive of an aromatic compound containing three or more closed rings, usually of the benzenoid type, e.g., sterols. See also polycyclic, nucleus (3).

polyol. A polyhydric alcohol, i.e., one containing three or more hydroxyl groups. Those having three hydroxyl groups (trihydric) are glycerols, those with more than three are called sugar alcohols, with general formula $\text{CH}_2\text{OH}(\text{CHOH})_n\text{CH}_2\text{OH}$, where n may be from 2 to 5. These react with aldehydes and ketones to form acetals and ketals.

See also alcohol, glycerol.

polyolefin. A class or group name for thermoplastic polymers derived from simple olefins, among the more important are polyethylene, polypropylene, polybutenes, polyisoprene and their copolymers. Many are produced in the form of fibers. This group comprises the largest tonnage of all thermoplastics produced.

polyorganosilicate graft polymer. An organoclay to which a monomer or an active polymer has been chemically bonded, often by the use of ionizing radiation. An example is the bonding of styrene to a polysilicate containing vinyl radicals, resulting in the growth of polystyrene chains from the surface of the silicate. Such complexes are stable to organic solvents. They have consid-

polypyrrolidone. Synonym for nylon-4.

"Polyrad."²⁶⁶ TM for reaction products of "Amine D" and ethylene oxide.

Grade: Various grades which differ in chain length of polyoxyethylene units and free amine content. Vary in viscosity at 25C from 0.5–24.8 poises.

Use: Corrosion inhibitors and detergents in petroleum processing equipment, wetting and emulsifying agents, inhibiting hydrogen chloride.

"Polyram."⁵⁵ TM for a wettable powder.

Hazard: Toxic by ingestion and inhalation.

Use: Fungicide and approved for many vegetables.

polysaccharide. A combination of nine or more monosaccharides, linked together by glycosidic bonds. Examples: starch, cellulose, glycogen.

See also carbohydrate, phycocolloid.

polysiloxane. See siloxane.

"Poly-Solv."⁸⁴ TM for a series of glycol ether solvents for paints, varnishes, dry cleaning soaps, cutting oils, insecticides.

D2M. Diethylene glycol dimethyl ether.

Use: Anhydrous reaction medium for organometallic syntheses.

polysorbate. (USAN name for a polyoxyethylene fatty acid ester). One of a group of nonionic surfactants obtained by esterification of sorbitol with one or three molecules of a fatty acid (stearic, lauric, oleic, palmitic) under conditions which cause splitting out of water from the sorbitol, leaving sorbitan. About 20 moles of ethylene oxide per mole of sorbitol are used in the condensation to effect water solution.

Properties: Lemon to amber, oily liquids; d 1.1; faint odor and bitter taste; most types are soluble in water, alcohol, and ethyl acetate. Combustible.

Grade: Polysorbate 20 (polyoxyethylene (20) sorbitan monolaurate). Polysorbate 60 (polyoxyethylene (20) sorbitan monostearate). Polysorbate 80 (polyoxyethylene (20) sorbitan monooleate). Polysorbate 65 (polyoxyethylene (20) sorbitan tristearate).

Use: Surfactant, emulsifying agent, dispersing agents, shortenings and baked goods, pharmaceuticals, flavoring agents, foaming and defoaming agents.

See also sorbitan fatty acid ester.

polystyrene. CAS: 9003-53-6.

($C_6H_5CHCH_2$)_n. Polymerized styrene, a thermoplastic synthetic resin of variable molecular weight depending on degree of polymerization.

Properties: Transparent, hard solid; high strength and impact resistance; excellent electrical and

thermal insulator. Attacked by hydrocarbon solvents but resists organic acids, alkalies, and alcohols. Not recommended for outdoor use, unmodified polymer yellows when exposed to light, but light-stable modified grades are available. Easily colored, molded, and fabricated. Copolymerization with butadiene and acrylonitrile and blending with rubber or glass fiber increase impact strength and heat resistance; autoign temperature 800F. Combustible.

Derivation: Polymerization of styrene by free radicals with peroxide initiator.

Forms: Sheet, plates, rods, rigid foam, expandable beads or spheres.

Hazard: As for foam, plastic.

Use: Packaging, refrigerator doors, air conditioner cases, containers and molded household wares, machine housings, electrical equipment, toys, clock and radio cabinets. (As foam): thermal insulations, light construction as in boats, etc., ice buckets, water coolers, fillers in shipping containers, furniture construction. (As spheres): radiator leak stopper.

See also "Styron", "Styrofoam."

polysulfide elastomer. A synthetic polymer in either solid or liquid form obtained by the reaction of sodium polysulfide with organic dichlorides such as dichlorodiethyl formal, alone or mixed with ethylene dichloride. Outstanding for resistance to oils and solvents and for impermeability to gases. Poor tensile strength and abrasion resistance but are resilient and have excellent low-temperature flexibility. Some grades have fairly strong odor, which is not objectionable in most applications. Sealant grades are furnished in two parts which cure at room temperature when blended.

Use: Gasoline and oil-loading hose, sealants and adhesive compositions, binder in solid rocket propellants, gaskets, paint spray hose.

See also "Thiokol."

polysulfone. A synthetic thermoplastic polymer. Properties: Hard, rigid, transparent solid. Tensile strength 10,000 psi, d 1.24, flexural strength 15,000 psi, good electrical resistance, minimum creep, low expansion coefficient. Soluble in aromatic hydrocarbons, ketones, and chlorinated hydrocarbons; resistant to corrosive acids and alkalies, to heat and oxidation and to detergents, oils, and alcohols. Dimensionally stable over temperature range -100 to +148C; tends to absorb moisture, readily processed and fabricated. Combustible, but self-extinguishing.

Derivation: Condensation of bis-phenol A and dichlorophenyl sulfone.

Use: Power-tool housings, electrical equipment, extruded pipe and sheet, auto components, elec-

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eralocorticoids (which affect mineral metabolism), the glucocorticoids (which affect glucose metabolism), the adrenal cortical androgens (which control secondary sex characteristics in males), and the estrogens (estrus-producing hormones).

Secretion of the glucocorticoids is controlled by adrenocorticotropin (ACTH), secreted by the anterior lobe of the pituitary. Secretion of the mineral corticoid is ultimately controlled by the kidney secretion of renin. See ANDROGEN; ESTROGEN; PITUITARY GLAND; STEROID. [H.Lip.]

Malfunctions of the adrenal glands may be divided into two major categories, those of the medulla and those of the cortex. The principal disorders of the medulla are tumors. One rare type, the pheochromocytoma, is an actively secreting neoplasm which causes excessive production of the medullary hormones, epinephrine and norepinephrine. Other rare, nonsecreting tumors of the medulla are the ganglioneuroma and the highly malignant neuroblastoma of infancy and childhood.

Congenital defects of the adrenal glands are principally those related to the absence of glandular tissue or to the lack of its normal secretory ability (hypoplasia).

The adrenals are susceptible to certain infections, largely because they are so well vascularized. The resulting inflammations may produce acute reactions and crises, or in lesser cases, may eventually cause scarring and other degenerative changes with or without hormonal alteration.

There are many varieties of neoplasms of the adrenal cortex. They may be either primary or metastatic. Either type may cause alterations of adrenal hormone output, and the classification is often based on clinical findings and sometimes on histologic types. Major clinical categories include Cushing's syndrome, the adrenogenital syndrome, feminizing syndromes, hyperaldosteronism, and combinations of components of any or several of these.

The functional types, in which hormone output is altered, are more readily recognized because of the changes they effect. Nonfunctional tumors may be silent and therefore often offer a problem in diagnosis. The most common metastatic malignancies in the adrenals arise from bronchogenic carcinoma, although other carcinomas may also spread to these glands. See ADDISON'S DISEASE. [E.G.St./N.K.M.]

Adsorption The property of an interface between two immiscible phases (solid, liquid, or vapor) to attract and concentrate components of either phase or both phases as an adsorbed interfacial film. Adsorption is a basic thermodynamic property of interfaces, resulting from a discontinuity in intermolecular or interatomic forces. It is also important in nearly all industrial processes and products.

Some definitions that describe adsorption are as follows: The adsorbent is the solid or liquid which adsorbs. The adsorbate is the solid, liquid, or gas which is adsorbed as molecules, atoms, or ions. Physical adsorption or physisorption is reversible adsorption by weak interactions only; no covalent bonds occur between the adsorbent and adsorbate; heats of physical adsorption are usually less than 15–20 kcal/mole (63–84 kilojoules/mole). Chemical adsorption or chemisorption is adsorption involving stronger interaction between adsorbate and adsorbent usually accompanied by rearrangement of atoms within or between adsorbates; reaction occurs between the surface of the adsorbent and the adsorbate; heats of chemisorption are usually in excess of 20–30 kcal/mole (84–126 kilojoules/mole).

Nearly all vapors tend to adsorb onto inorganic solids at temperatures not too much above their boiling point. The intermolecular attractive forces which cause the physical adsorption of vapors are generally dominated by the London dispersion forces, an attraction caused by the perturbation of electron orbits by adjacent atoms. Another attractive force important in vapor adsorption is the interaction of electron-donor (basic)

sites of vapor molecules with electron-acceptor (acidic) sites of adsorbents, or vice versa. These short-range attractions are much stronger than dipole interactions. Silica, an acidic adsorbent, adsorbs basic vapors (water, ammonia, and so forth) much more strongly than acidic vapors (chloroform, CO₂, NO₂, and so forth) regardless of the dipole moments.

The adsorption of water is dominated by hydrogen bonding, an intermolecular acid-base interaction onto neutral surfaces such as graphite or polyethylene, except for the acidic or basic sites provided by impurities on these neutral surfaces.

The strong interactions of chemisorption lead to surface compounds with various degrees of covalent bond character. The adsorbed layers are only one molecule thick because covalent bonds exist only between adjacent atoms. Chemisorption occurs on metals and semiconductors and on oxides and sulfides, but is most often observed on transition metals such as silver, nickel, cobalt, platinum, rhodium, and tungsten. Chemisorption is a necessary step in catalysis by these materials. See CHEMICAL DYNAMICS; INTERMOLECULAR FORCES.

Heterogeneous catalysis, in which gas or liquid reactants are specifically adsorbed to a dissimilar phase and chemically altered during their brief retention time, is basic to many industrial processes in the petrochemical, polymer, and chemical industries.

Purification by adsorption is perhaps the oldest known application; examples are wine and beer clarification, color removal in sugar processing, industrial wastewater treatment, and toxic gas adsorption in gas masks.

Adsorption is the basic phenomenon of chromatographic separations, which separate and concentrate components of mixtures according to strength of adsorption onto adsorbents in chromatographic columns.

Adsorption of surface-active substances is the key process in the use of soaps, detergents, emulsifiers, wetting agents, dyes, lubricants, and surface treatments. Other industries dependent on adsorption processes include agriculture, mining, petroleum recovery, papermaking, printing, and photography. See CATALYSIS; CHEMICAL SEPARATION TECHNIQUES; CHROMATOGRAPHY; RESPIRATOR. [F.M.F.]

Aegyptopithecus A primate that lived during Oligocene times, 30,000,000 years ago, in the Egyptian Fayum (see illustration), and is believed to be the common ancestor of humans and apes. The names *Aegyptopithecus zeuxis* and "dawn ape" were given by Elwyn Simons.



Artist's rendering of *Aegyptopithecus*. (Duke University)

Aegyptopithecus had an estimated weight ranging from 8 to 12 lb (3.6 to 5.4 kg). The cranial capacity was about 1.8 in.³ (30 cm³) larger than that of any of its mammalian contemporaries, relative to body size. The teeth were equipped for a

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anions of strong acids with HCO_3^- species to yield an increase of m alkalinity. Thus, the cation exchanger can again replace cations by protons (Fig. 6.7). Because of the limited efficiency of carbonic acid, only partial regeneration of both resins is achieved. As a consequence, the exchange on both resins remains incomplete, yielding considerable leakage of both anions and cations. The possible development of the chemical state of the water is plotted in Figure 6.7.

7. Flocculation

7.1. Introduction

Most surface waters contain finely dispersed substances that cannot be separated completely by simple sedimentation or rapid filtration. These dispersed particles are responsible for the turbidity of water and may consist of minerals such as silica and clays, algae cells, bacteria, viruses, organic detritus, or insoluble pollutants [7.1]. In groundwater, particles can be formed by a preceding precipitation (e.g., lime precipitation for hardness removal) or by oxidation of iron(II) and manganese ions. The finely dispersed particles can be aggregated to form removable flocs by flocculation techniques using suitable chemicals known as coagulants and flocculants.

The aggregates formed can be removed to a wide degree by subsequent separation processes such as sedimentation, flotation, or filtration. In the production of high-quality water such as drinking water, sedimentation and flotation usually are followed by rapid filtration, while direct filtration of flocs (see Chap. 8) is limited to raw water with low suspended solids and coagulant demand.

The removal of *particulate substances* is usually the primary objective of flocculation, but important cases exist in which *dissolved* inorganic and organic substances must be removed effectively by coagulants and flocculants via precipitation, coprecipitation, or adsorption onto the flocs. Examples are phosphates, some heavy metals, or dissolved organic substances of natural or anthropogenic origin. In view of the new standards for lower concentrations of organic substances in drinking water, the removal of DOC has become a major task in water treat-

ment, especially in raw water colored by natural humic substances or having a high DOC [7.2].

7.2. Definitions

Scientific and technical terms that are used frequently in flocculation are listed and explained below [7.3]–[7.6]:

Colloids are particles with diameters in the range of $0.001\text{--}1\text{ }\mu\text{m}$. They do not settle, and they cause turbidity. Their high specific surface areas induce important interfacial forces and phenomena between the colloid particles.

Suspended particles have diameters $>1\text{ }\mu\text{m}$ and are detected under a light microscope. They may be removed by settling if their density is greater than that of water.

Aggregation denotes the general process of forming larger flocs from primary particles.

Stabilization. Colloidal and suspended particles can be stabilized against aggregation. Stabilization is caused by van der Waals attractive forces, by the electrostatic surface charge of the particles, by an adsorbed layer of polymers, or by forces of hydration. Most important in aqueous systems are the negative surface charges formed by the dissociation of protons from hydroxyl functions belonging to silica groups or from acidic carboxylic functions [7.1], [7.3], [7.4].

Destabilization. Stable particles are rendered unstable through neutralization of their negative surface charges by adsorption of positively charged coagulants.

Coagulation refers to (1) the reduction or elimination of electrostatic repulsion forces between particles via addition of certain coagulants, and (2) in technical terms, the first phase of floc formation after chemical mixing and destabilization, but before dosing of flocculants (see below) [7.4].

Flocculation. (1) Flocculation is the mechanism of floc aggregation by high molar mass polymers that adsorb on the particles and form bridges between them. (2) In water treatment, flocculation is the phase of formation of large

flocs by addition of a secondary chemical (after addition of the coagulant) called a flocculant or flocculant aid [7.4]. (3) The term can be used for the entire water treatment process (coagulation plus flocculation), as in this chapter. In English-speaking countries, the terminology "coagulation and flocculation" is also used frequently to describe this process.

Coagulants are chemicals used for destabilization in the first phase of floc formation. Most common are the salts of Al^{3+} and Fe^{3+} ions; sometimes, organic cationic polymers are used.

Flocculants designate chemicals used to improve the formation of larger flocs. Flocculants are high molar mass water-soluble polymers. Flocculant aid or coagulant aid is used as a synonym.

Precipitation is the formation of insoluble substances from dissolved matter and the chemicals added. The important coagulants Al^{3+} and Fe^{3+} salts can precipitate as hydroxides in the neutral pH region and are thus removed from water.

Coprecipitation is the inclusion of dissolved or particulate substances into a precipitate (e.g., of aluminum or iron(III) hydroxide). Particles are then aggregated by the important *sweep coagulation mechanism*, with the amorphous, gelatinous hydroxide flocs acting as particle collectors.

7.3. Chemicals Used in Coagulation and Flocculation

7.3.1. Inorganic Chemicals

(→ Flocculants, A11, pp. 251–252)

The most important coagulants in water treatment are the salts of iron(III) and aluminum ions, available in various commercial forms and purities. Iron(III) chloride or aluminum sulfate (alum) is used frequently because of low cost. Mixed coagulants are also available, especially for applications in wastewater treatment. These coagulants dissolve readily in water, and the metal ions form hexaquo complexes, $\text{Me}(\text{H}_2\text{O})_6^{3+}$, which are acidic species and give up protons [7.7].

The aquo complexes are stable below pH 3–4 (Al^{3+}) and 1–2 (Fe^{3+}). After stepwise formation of hydroxo-aquo complexes with increasing pH, insoluble hydroxides are formed at higher pH (Al^{3+} , pH > 5.5–6.0; Fe^{3+} , pH > 4.5–5) as voluminous, amorphous flocs. The residual concentrations of dissolved metal ions are well below the drinking water standards of 0.2 mg/L for Fe and Al. The $\text{Al}(\text{OH})_3$ dissolves as aluminate $[\text{Al}(\text{OH})_4]^-$ at pH > 7.5–8.0, which must be avoided in water treatment [7.8]; $\text{Fe}(\text{OH})_3$ redissolves at pH > 10–11 to form $[\text{Fe}(\text{OH})_4]^-$.

The cationic partially hydrolyzed species present in a slightly acidic medium can condense to form polymeric products with a high positive charge. Various oligomeric and polymeric products are known [e.g., $\text{Al}_{13}(\text{OH})_{32}^{7+}$ and $\text{Fe}_3(\text{OH})_4^{5+}$]. For more details on hydrolysis and condensation reactions, see [7.7]. Cationic aluminum polymers are available as commercial products or can be produced by partial hydrolysis and polymerization. These coagulants are usually called polyaluminum salts, but their composition and degree of hydrolysis vary widely.

Iron(III) and aluminum salts exhibit two distinctly different mechanisms in particle aggregation [7.9]:

- 1) Cationic polymeric species adsorb on negatively charged particles and neutralize them at an optimum dosage (charge and neutralization). Overdosing leads to restabilization because of charge reversal.
- 2) Insoluble hydroxides precipitate and enmesh the particles by the sweep coagulation mechanism. Optimum coagulation is achieved under conditions of maximum precipitation of the hydroxides. The nature and concentration of particles have no effect.

The sweep coagulation process is the dominant mechanism in most cases, whereas charge neutralization may be important in the coagulation of algae, humic colloids, or more concentrated suspensions and at a pH of < 6.0–6.5.

The optimum dosages of Al^{3+} or Fe^{3+} may vary with water source and, because of frequent changes in raw water quality, even in the same plant within a few hours. The typical dosages given below are a preliminary guide for frequent cases:

- 1) Treatment plants for river water with settling: 0.05–0.2 mol/m³ Me^{3+}

- 2) Treatment plants for lake and impounded water with direct or in-line filtration: $0.01 - 0.1 \text{ mol/m}^3 \text{ Me}^{3+}$
- 3) Treatment plants for color and DOC removal: $0.01 - 0.05 \text{ mmol Me}^{3+}$ per milligram DOC; optimum pH values for Al^{3+} : $5.5 - 6.5$; for Fe^{3+} : $4.5 - 6$ [7.10], [7.11]

Laboratory tests are usually performed to find the most suitable coagulation conditions before adjusting dosages in the plant.

Other inorganic chemicals are much less common, such as "activated silica," a polymeric flocculant, or $\text{Mg}(\text{OH})_2$, formed at $\text{pH} > 10.5$ [7.5].

7.3.2. Organic Chemicals

(→ Flocculants, A11, pp. 252–253)

Organic substances added in flocculation are linear, water-soluble polymeres (synthetic, natural, or modified natural polymers) with mean molar masses in the range of $10^4 - 10^7 \text{ g/mol}$. The following monomers are included [7.5]:

Acrylamide (nonionic), acrylic acid (anionic), *N,N*-(dimethylaminopropyl)methacrylate (cationic), and ethyleneamine (cationic).

The resulting polymers can be nonionic, cationic, or anionic; the charge density can vary from low to very high, depending on copolymer composition. The molecular diameter of the polymers in solution is $10 - 500 \text{ nm}$.

Most commercial polymers are copolymers, containing at least two monomer types. The monomer content of these products is limited to $0.05 - 0.1\%$ if they are used in drinking water treatment [7.12].

Highly charged cationic polymers can be applied as coagulants, instead of Al^{3+} and Fe^{3+} salts because of their effective charge neutralization mechanism at optimum dosage [7.13]. The dosages required can be rather high ($1 - 10 \text{ g/m}^3$).

High molar mass polymers with low to medium charge densities are flocculants, acting by the adsorption-bridging mechanism [7.3]. Typical dosages are in the range of $0.05 - 1.0 \text{ mg/L}$, and the polymer should be added at least 30 s after addition of the metal salts. Flocs formed with the aid of these polymers are larger, are more resistant to erosive and destructive forces, and settle faster.

In addition to synthetic polymers, natural or modified natural polymers such as starch, modi-

fied starch, or alginates can be used. Since natural polymers tend to be less effective, are unstable, and may support bacterial growth, they must be applied with more care.

7.4. Kinetics of Aggregation

The particle transport processes necessary for particle collisions are usually slower than mixing, chemical reactions (e.g., hydrolysis), adsorption, and destabilization. The kinetic rate of aggregation is thus determined by the particle collision rate, which itself depends on three basic transport processes [7.3], [7.14].

Particle diffusion is based on Brownian movement and dominates in concentrated colloidal systems if the particle diameter is $< 0.5 - 1 \mu\text{m}$. This *perikinetic coagulation* is probably not important in water treatment or is limited to the first short phase until microflocs appear with diameters $> 1 \mu\text{m}$.

Hydrodynamic shear gradients imposed by stirring or turbulent flow are responsible for different particle velocities. This important collision mechanism (*orthokinetic coagulation*) can be described for the initial period by

$$\ln N/N_0 = -4/\pi \cdot \alpha \cdot \phi \cdot \bar{G} \cdot t \quad (7.1)$$

where N and N_0 are the total particle concentrations at time t or $t = 0$; α is the collision efficiency factor; \bar{G} is the mean shear gradient (s^{-1}); ϕ is the volume concentration of solids; and t is time.

The collision efficiency factor depends on the extent of destabilization of the particles (chemical factor), while \bar{G} represents shear conditions. The mean value \bar{G} is used for turbulent conditions and can be calculated [7.3] according to the following:

For back-mix reactors

$$\bar{G} = \left(\frac{P}{\eta \cdot V} \right)^{\frac{1}{2}} \quad (7.2)$$

where P is the power input of stirrer; V the reactor volume; and η the dynamic viscosity.

For pipes and channels

$$\bar{G} = \left(\frac{\Delta p \cdot v}{l \cdot \eta} \right)^{\frac{1}{2}} \quad (7.3)$$

where Δp is the pressure loss; v the flow rate; l the length of the pipe; and η the dynamic viscosity.

The orthokinetic equation is not longer valid if larger flocs are formed and destroyed again. In this case, the formula must be extended by a floc destruction term [7.15], describing the fact that larger flocs are formed at low \bar{G} values, while high \bar{G} values induce smaller flocs. In designing flocculation reactors, the use of two or more reactors can reduce overall reactor volume. The first reactor is operated at high \bar{G} values for high collision rates but small flocs, whereas the second reactor is needed to produce large flocs at low \bar{G} values (see Section 7.5).

The final flocs are separated by either settling, flotation, or filtration, whereby the different floc velocities induce further collisions and floc growth. This third transport process is significant in settling and flotation tanks.

7.5. Process Technology for Coagulation and Flocculation

The major process steps in coagulation and flocculation are listed in Table 7.1. Steps 1 and 2 are physically different processes but are quite rapid and usually occur in the same reactor, the rapid mix system. Steps 3 and 4 are performed in one, two, or more reactors, depending on plant design.

7.5.1. Dosing

Coagulants and flocculants are generally prepared as a stock solution or delivered as a concentrated solution. Some restrictions exist on the

concentration of stock solutions and their predilution before dosing. *Iron(III) and aluminum salts* may start to precipitate or hydrolyze at concentrations $<1\%$ (Fe, Al) and pH values $>1-2$. Polymeric aluminum salts should be diluted only to the ratios given by the producer. All metal salts are acidic or alkaline solutions and require safety precautions. Iron(III) salts are especially corrosive.

Organic polymer solutions exhibit high viscosities at concentrations $>0.2-0.5\%$, depending on their molar mass. Dissolution sometimes requires several hours, and the stock solution may not be highly turbulent because of loss of efficiency and should be stored for only a limited time.

National and upcoming international standards exist regarding the purity of all applied chemicals to avoid secondary pollution in the finished drinking water. Cationic polymers, especially, are not approved for use in several countries because their risks are unknown.

7.5.2. Rapid Mixing

The small flow of a dissolved coagulant and flocculant must be distributed rapidly in the large water flow by suitable reactors. Efficient, rapid mixing is very important for destabilization of colloids. The present state of knowledge and technology is summarized in [7.16]. Rapid mixing reactors can be designed as plug-flow or back-mix systems. The latter apparently have some deficiencies in terms of coagulation, because the coagulant species may not be transported evenly to all particles in a short time. Mixing studies indicate that the metal salts should be mixed completely within $0.1-1$ s, which may be difficult to achieve in large plants [7.16].

Possible rapid mixers are listed in Table 7.2. If polymeric flocculants are dosed after the primary coagulants (usually metal salts), the retention time between dosing points should be at least 30 s or more to avoid a higher polymer demand. Polymers should not be mixed with excessive intensity to limit microfloc destruction and polymer breakage.

7.5.3. Reactors for Floc Formation

A variety of reactors for aggregation exist in water treatment, depending on historical developments, national regulations and experience,

Table 7.1. Process steps for coagulation and flocculation systems

Step	Task
Dosing of coagulants and rapid mixing	homogeneous distribution of chemicals
Destabilization	adsorption of coagulants on colloids and suspended particles, precipitation of dissolved substances
Aggregation to microflocs (coagulation)	rapid formation of small flocs from destabilized particles in high shear gradients, without flocculants (polymers)
Aggregation to macroflocs (flocculation)	formation of removable large flocs, with or without a flocculant (polymer)